THE ELASTIC AND VISCOELASTIC PROPERTIES OF RIGID BODIES IN CONTACT

A Senior Scholars Thesis

by

MOHAMED BENHALIMA

Submitted to the Office of Honors Undergraduate Research
Texas A&M University
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Approved by:

Research Advisor: Zachary Grasley
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ABSTRACT

The Elastic and Viscoelastic Properties of Rigid Bodies in Contact. (May 2012)

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The process by which to utilize an atomic force microscope (AFM) to measure the viscoelastic properties of a given material through indentation experiments is implemented. The difficulties faced regarding both the data modeling and acquisition are discussed. Analytical viscoelastic solutions can generally be used for high indentation forces in stiff materials. However, softer materials require the usage of numerical routines in order to evaluate low forces of contact. Previous modeling of the indentation problem has been shown to under predict the stiffness of the material. A correction in geometry has been implemented in the numerical routine, and this change provides modeling of AFM indentation data with sufficient accuracy.

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CHAPTER I

INTRODUCTION

Construction materials such as concrete and other cementitious materials include a complex structure that is composite in nature on the nanometer length scale. In order to directly measure the properties of individual distinct phases, it is necessary to interact with individual phases. Nanoindentation has been a popular technique for measuring the mechanical constitutive properties of small phases in many classes of materials, including construction materials (Grasley and Jones, 2011). However, traditional nanoindenters actually probe properties on the micron length scale. The development of quantitative indentation measurement capabilities of the atomic force microscope (AFM) now allows one to interact with individual phases on the nanometer length scale. Accompanying the advantages of the ability of the AFM to probe nanometric phases are challenges associated with measurements on very small length and force scales. Thus, traditional analysis tools utilized for extracting mechanical constitutive properties from traditional nanoindentation experiments are not always applicable to indentation results obtained from an AFM (Grasley and Jones, 2011).

This thesis follows the style of *Journal of Materials and Structural Integrity*.

Another challenge when dealing with cementitious materials is the fact that these materials are not fully conservative. More specifically, cementitious materials are known to be viscoelastic on the macroscale, and the macroscale viscoelastic response is often attributed to the response of the nanometric calcium silicate hydrate (Grasley and Jones, 2011). The objective of the study described herein was to develop a systematic means for measuring the viscoelastic properties of nanometric phases – specifically the C-S-H phase within concrete – using an AFM. While several researchers have utilized AFM to quantify both elastic and viscoelastic properties of materials, an evaluation of the use of analytical or numerical solutions for analyzing AFM indentation data for viscoelastic property extraction is not presently available. In this paper, analytical and numerical approaches for modeling time-dependent AFM indentation of viscoelastic materials are presented and advice is given on the appropriate use of each approach for accurate extraction of material properties (Grasley and Jones, 2011).

CHAPTER II

METHODS

The Hertz model

The indentation experiments have traditionally been modeled using an approach developed by Hertz. This method yields the following relationships between indentation depth and force (Hertz, 1881):

$$F(t) = \frac{4}{3} M R^{1/2} h_0(t)^{3/2}, \tag{1}$$

where R represents the tip radius of the indenter in contact with an infinite plane, h_0 is the indentation depth, t is elapsed time, and the indentation modulus M is defined by the following equation:

$$M \approx \frac{E}{1-\nu^2}. \tag{2}$$

The indenter is assumed to be a perfectly rigid body. In addition, the material being indented is considered isotropic and linear with a Modulus of Elasticity E and a Poisson's ratio ν. In order to account for the conical shape of the indenter being used and the significant surface adhesion of the materials (Sneddon, 1965; Dejagun and Muller, 1975; Johnson and Kendall, 1971), the Hertz equation has been modified to:

$$F(t) = \frac{2\tan(\phi)}{\pi} M h_0(t)^2 , \tag{3}$$

where ϕ is a half of the angle of indenter tip of conical shape. The principle of elastic-viscoelastic correspondence was used to obtain a Hertz' solution to account for the linear material being indented (Read, 1950; Graham, 1965; Graham, 1967; Vandamme and Ulm, 2006). It is worthwhile to note that contact problems derived from the correspondence solution are applicable only to areas of contact that increase with time.

If we assume that the Poisson's ratio ν is independent of time, $H(t)$ is the Heaviside function, and F_0 is the applied force's magnitude (Jones and Grasley, 2011), then Eq.(2) becomes,

$$h_0(t) = \left(\frac{\pi\left(1-\nu^2\right)}{2\tan(\phi)} F_0 J(t) \right)^{1/2} , \tag{4}$$

where $J(t)$ is the compliance of the material. Eq. (3) can be used with sufficient accuracy to fit the creep data obtained in an indentation experiment.

The assumptions associated with the Hertz solution

There are numerous assumptions associated with Eq. (4); these include negligible forces of attraction, small deformations, and linearity with respect to constitutive properties. (Attard, 2007). Furthermore, the analytical expressions like (4) are subject to geometry restrictions ((Grasley and Jones, 2011).

In order to address the problem of assumptions associated with surface forces as well as geometric restrictions, Attard devised a numerical routine that can be used to model elastic and viscoelastic indentations with sufficient accuracy (Attard and Parker, 1992; Attard 2007). For a rigid indenter with cylindrical or conical geometry and deforming a planar surface of a given material, the following relationship applies:

$$h(r) = h_0(r) - u(r), \tag{5}$$

where $h(r)$ is the vertical distance between the indenter and the surface of the material, $h_0(r)$ is the rigid separation that applies for perfectly rigid material and indenter, $u(r)$ is the deformation in the vertical direction of the surface of the material being indented, and r is the radial coordinate. The term $h_0(r)$ can be calculated using the following equation (Grasley and Jones, 2011):

$$h_0(r) = h_0(r=0) + C(r), \tag{6}$$

where $C(r)$ is a constant determined by the shape of the tip. The displacement in the vertical direction depends on the constitutive properties and the pressure exerted on the surface. The pressure can be either attractive or repulsive, and it increases as the tip approaches the surface. For a viscoelastic material, one can write:

$$u(r,t) = \int_0^t \frac{W(t-t')}{\pi} \int_\Omega \frac{\partial p(h(r',t'))}{\partial t' |r-r'|} dr' dt'$$
$$\text{or} \tag{7}$$
$$\frac{\partial u(r,t)}{\partial t} = \frac{W(t)}{\pi} \int_\Omega \frac{\partial p(h(r',t))}{\partial t |r-r'|} dr'$$

where t' and r' are dummy time and radial variants respectively; $p(h(r,t))$ is the pressure that the indenter imposes on the material as the separation between them shortens. The expression $p(h(r,t))$ depends on the material properties of both the indenter and the material being indented as well as the repulsive or attractive nature of the interaction. Most materials exhibit moderate surface attraction levels that switch to repulsive forces as the separation between the two bodies becomes smaller.

The numerical and analytical approaches

For the purpose of generating a curve of $h_0(r=0)$ versus t using the expressions above, a self-consistent routine was developed using Matlab. This routine consists of stepping $h_0(r=0)$ at a rate that approaches that used in the lab experiment until the required interpenetration between the indenter and the surface is achieved. Eq. (5) is used at each step to provide an approximation of the new deformation corresponding to the current $h_0(r)$ in addition to the $h(r)$ from the preceding step. Also, the pressure profile is calculated at each step using (Grasley and Jones, 2011):

$$\frac{\partial p(h(r,t))}{\partial t} = \frac{\partial p(h(r,t))}{\partial h}\frac{\partial h}{\partial t} \approx \frac{\partial p(h(r,t))}{\partial h}\frac{1}{\Delta t}\left[\left(h_0(r,t_n)-h_0(r,t_{n-1})\right)-\left(u(r,t_n)-u(r,t_{n-1})\right)\right] \tag{8}$$

where n is the actual time step equal to Δt. In addition, the new rate of $u(r,t)$ is estimated with Eq.(6). Finally, $u(r,t)$ is compared to the estimated $u(r,t)$ using an error reduction algorithm; if the difference between the two quantities is relatively large, both quantities are blended and the numerical routine starts overs by taking the blended value as the new calculated quantity. The process is done repetitively until the difference is minimized to an acceptable value. Once this goal is achieved, $h_0(r=0)$ is stepped up to a new value and this is repeated until experimental force values equal the simulated quantities for the force. All the steps are taken to be relatively small when compared to the depth of indentation; this constraint is necessary for the convergence of the calculations (Grasley and Jones, 2011).

CHAPTER III

RESULTS

The numerical modeling method defined in the previous section is a constitutive law that provides a correlation between the vertical distance between two bodies and the pressure on the surfaces of those bodies. However, the pressure should be a function of the closest distance between the bodies rather than simply the vertical distance. This is mainly due to the fact that the tips utilized in AFMs are extremely sharp, and therefore the vertical distance does not describe the correct behavior. Therefore, it is essential to update the numerical routine described in the previous section to the following:

$$\frac{\partial u(r,t)}{\partial t} = \frac{W(t)}{\pi} \int_{\Omega} \frac{\partial p(h_c(r',t))}{\partial t |r-r'|} dr', \tag{9}$$

where $h_c(r,t)$ is the shortest distance between the surface and the indenter at a given radial position r. The closest distance is then easily calculated using the formula:

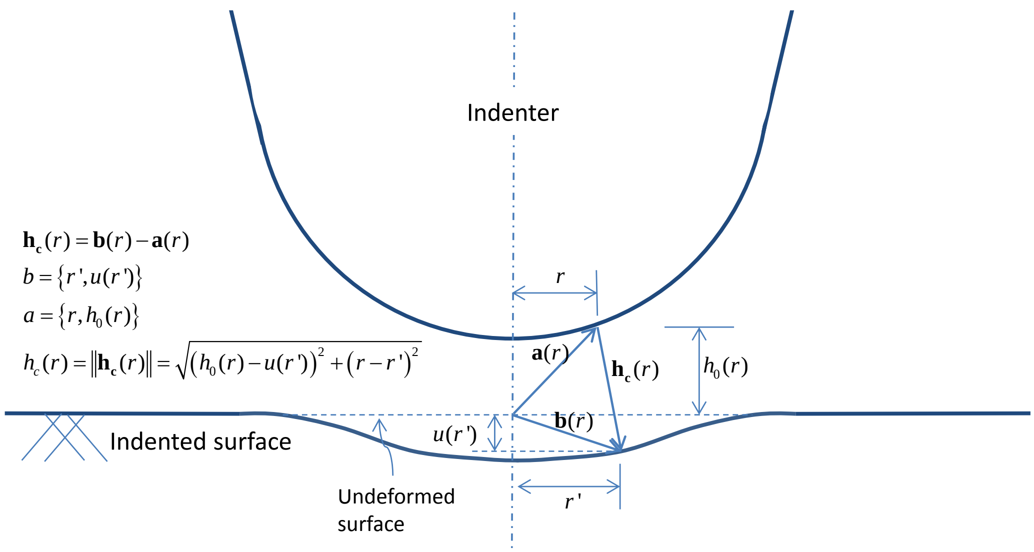
$$h_c(r) = \min\left[\sqrt{\left(h_0(r,t) - u_z(r',t)\right)^2 + (r-r')^2} \right] \quad \forall r' \in \{0,\infty\}, \tag{10}$$

As shown in Figure 1, the error in the calculation of the surface pressure resulted by using h instead of h_c was determined. In the simulations performed for this analysis, the indenter characteristics were chosen to be cone angles of 30° or 60°, an indentation modulus ranging between 0.4 and 40 GPa, and a constant tip radius of 5nm. The pressure of interaction was defined as (Grasley and Jones, 2011):

$$p(h_c(r)) = p_r \exp\left(-\kappa h_c(r)\right) + p_r \left(\frac{z_0}{h_c(r)}\right)^{1.5}, \tag{11}$$

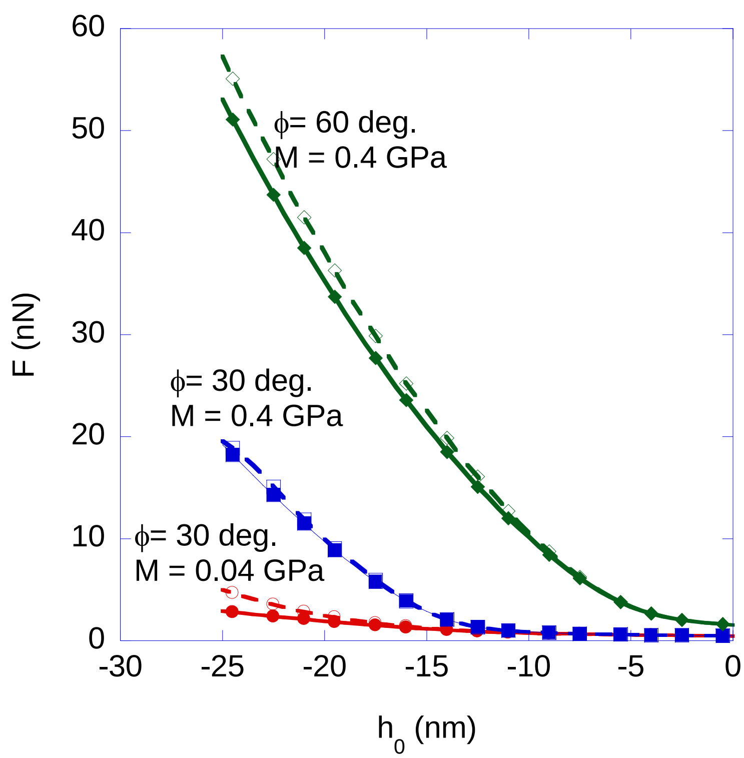
where z_0, κ, and p_r are constants defining the shape of the function.

Figure 1 Illustration of the calculation of the shortest separation distance (h_c)



Using the data of the numerical routines, a force (F) vs. rigid separation (h_0 at $r=0$) curve was obtained for each different simulation and the results are illustrated in Fig.2. For each simulation, the usage of h instead of h_c results in an incorrect under prediction of the modulus of elasticity (stiffness) of the material being indented. The largest error is observed when using the sharpest tip in addition to the most compliant (soft) material. At the deepest indentation level, the slope errors range from 86-100%. Even though the effect of considering h_c is negligible for shallower depths, there is a great benefit acquired when implementing it for higher depths, which are often used for stiff materials such as calcium silica hydrate (C-S-H). However, since computing h_c requires negligible additional calculation efforts, it is recommended to take the benefit from these numerical methods regardless of the material or the parameters considered for the experimental procedures (Grasley and Jones, 2011).

Figure 2 The force (F) versus rigid body separation ($h_0(r=0)$) plotted at each simulation



Even though the analytical approach seems to be simpler than the numerical method, the latter provides better accuracy in terms of the properties of the indented materials. Jones et al. (Jones and Ohlhausen, 2011) implemented a ratio of the attractive force to the repulsive force resulted during the indentation process. The ratio is expressed as follows for an elastic material:

$$\theta = \frac{M h_{0\max}{}^{3/2}}{3\gamma R^{1/2}}, \tag{12}$$

where $h_{0\max}$ is the deepest indentation level in the test and γ is the interfacial tension between the surface of the material and the indenter (Grasley and Jones, 2011).

For a viscoelastic material, the ratio of the attractive force to the repulsive force becomes time dependent such that:

$$\theta(t) = \frac{\int_0^t M(t-t')\left(\frac{\partial h_0(t')}{\partial t'}\right)^{3/2} dt'}{3\gamma R^{1/2}}. \tag{13}$$

When using s creep test, Eq.(5) may be reduced to simply:

$$\theta = \frac{M(t_f) h_{0\max}{}^{3/2}}{3\gamma R^{1/2}}, \tag{14}$$

where t_f is the entire duration of the creep test.

Jones et al. (Jones and Ohlhausen, 2011) proved that for $\theta > 100$, the error resulting of the usage of analytical expressions is negligible when the elastic properties are obtained.

CHAPTER IV

SUMMARY

Atomic force microscopy is extremely beneficial in terms of extracting viscoelastic properties of materials undergoing indentation tests. Numerical methods are used in fitting the experimental force and rigid displacement data obtained by AFM. On the other hand, analytical methods can be utilized to produce viscoelastic solutions with an acceptable level of accuracy. This research provides the proper guidelines to make a decision on whether to use numerical or analytical approaches. Furthermore, it implements the necessary modifications needed for traditional Attard numerical approach to account for the closest separation between the bodies involved in the indentation process (Grasley and Jones, 2011). This approach helps in avoiding the underestimation resulted by using the vertical separation only to calculate the material stiffness. Finally, the experiments conducted on time-dependent estimations still have numerous challenges to overcome. These challenges are discussed in addition to the necessary actions needed to improve the quality of the experimental results.

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